

# **Georgia Department of Natural Resources**

Environmental Protection Division Laboratory

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SOP 1-054 Rev. 1

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## **Waste Dilution – Method SW846-3580A**

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### **1 Scope and Application**

- 1.1 Method SW846-3580A is used to dilute of organic analytes in non-aqueous waste samples for methods SW846-8081A and SW846-8082. The samples are diluted in 10mL of hexane then analyzed by injection into a temperature programmable gas chromatograph with an electron capture detector. Identifications are obtained by analyzing a standard curve under identical conditions used for samples and comparing resultant retention times. Concentrations of the identified components are measured by relating the response produced for that compound to the standard curve response.
- 1.2 This method is restricted to analysts who have completed the requirements of the Initial Demonstration SOP. Refer to Section 13.1.

### **2 Definitions**

- 2.1 Refer to Section 3 and Section 4 of the Georgia EPD Laboratory Quality Assurance Manual for Quality Control definitions.
- 2.2 Refer to GA EPD Laboratory SOP – Organics Data Validation, SOP 1-052, online revision.

### **3 Interferences**

- 3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baselines in chromatograms.
- 3.2 Glassware must be scrupulously cleaned with hot water and detergent followed by de-ionized water then rinsed with methanol followed by acetone.
- 3.3 The use of high purity reagents and solvents is absolutely necessary to minimize interference problems.

3.4 Interfering contamination may occur when a sample containing low concentrations of analytes is analyzed immediately following a sample containing relatively high concentrations of analytes.

3.5 Matrix interferences may be caused by containments that are co-extracted from the sample.

#### **4 Safety**

4.1 Refer to Georgia EPD Laboratory Chemical Hygiene Plan, online revision.

#### **5 Apparatus and Equipment**

5.1 Scintillation Vials: 20mL with caps

5.2 Glass culture tubes: 5mL, 10mL & 25mL with caps

5.3 Volumetric flasks (Class A): 10mL

5.4 Disposable pipettes and bulbs

5.5 Glass Wool: baked at 400°C for 4 hours

5.6 Detergent: Steris Labklenz or equivalent

5.7 Brushes: various sizes

5.8 Syringe: 1mL

5.9 Spatula

5.10 Balance: Top loading, capable of accurately weighing to the nearest 0.01g

5.11 Balance: Analytical, capable of accurately weighing to the nearest 0.0001g

5.12 Vials: auto-sampler vials, clear and amber, screw top, 2mL, caps with septa and 300µL inserts

#### **6 Reagents and Standards**

6.1 Hexane: pesticide grade or equivalent

6.2 Acetone: pesticide grade or equivalent

6.3 Methanol: pesticide grade or equivalent

6.4 Sodium sulfate: granular, anhydrous, certified ACS grade suitable for pesticide residue analysis or equivalent, baked at 450°C for 4 hours

6.5 Refer to GA EPD Laboratory SOP – Organochlorine Pesticides in Waste by Gas Chromatography – EPA Method SW846-8081A, SOP 1-034, Rev. 6 or later for Pesticide Standards.

6.6 GA EPD Laboratory SOP – Polychlorinated Biphenyls (PCBs) in Waste by Gas Chromatography – EPA Method SW846-8082, SOP 1-036, Rev. 7 or later for PCB Standards.

#### **7 Sample Collection**

7.1 Refer to SW846-8000B, SW846-8081A & SW846-8082 for sample collection procedures.

#### **8 Calibration**

- 8.1 Analytical balances are serviced and calibrated once per year by an independent technician. Daily readings with certified weights are taken each morning to ensure calibration. A daily log is maintained with this information. All precision oven temperatures are measured with NIST approved thermometers and these measurements are recorded every morning in the daily temperature log.
- 8.2 Refer to GA EPD Laboratory SOP – Organochlorine Pesticides in Waste by Gas Chromatography – EPA Method SW846-8081A, SOP 1-034, online revision for Pesticide Calibrations.
- 8.3 GA EPD Laboratory SOP – Polychlorinated Biphenyls (PCBs) in Waste by Gas Chromatography – EPA Method SW846-8082, SOP 1-036, online revision for PCB Calibrations.

## **9 Quality Control**

- 9.1 Refer to GA EPD Laboratory SOP – Organochlorine Pesticides in Waste by Gas Chromatography – EPA Method SW846-8081A, SOP 1-034, online revision.
- 9.2 Refer to GA EPD Laboratory SOP – Polychlorinated Biphenyls (PCBs) in Waste by Gas Chromatography – EPA Method SW846-8082, SOP 1-036, online revision.

## **10 Procedure**

- 10.1 Create a batch consisting of a Blank and a Pesticide LCS/LCSD/MS/MSD (alternating between pesticide mixtures) and a batch consisting of a Blank and a PCB LCS/LCSD/MS/MSD and up to 20 samples. *Note: A single Blank can be used with both Pesticide and PCB batches if diluted on the same day.*
- 10.2 The Blank is defined as 10mL of hexane spiked with 1mL of surrogate solution.
- 10.3 The LCS/LCSD are each defined as 10mL of hexane spiked with 1mL appropriate QC spike containing surrogates.
- 10.4 The MS/MSD are each defined as 10mL of sample matrix diluted in hexane spiked with 1mL of appropriate QC spike containing surrogates.
- 10.5 Remove sample bottles or containers and standards from cold storage and allow equilibrium to room temperature prior to sample preparation, typically two hours.
- 10.6 Start the dilutions with the Blank, Pesticide LCS/LCSD and PCB LCS/LCSD.
- 10.6.1 Use a 10mL volumetric flask for each Blank and LCS QC sample. Fill each flask with at least 5mL, but no more than 8mL, of hexane.
- 10.6.2 Spike the Blank with 1mL of the Pesticide/PCB Surrogate Standard and bring up to 10mL volume with hexane and cap.
- 10.6.3 Spike the Pesticide LCS/LCSD with the appropriate Pesticide QC spike containing surrogate and the PCB LCS/LCSD with the PCB QC spike containing surrogate and bring each up to 10mL volume with hexane and cap.

- 10.7 Samples may be diluted in 10mL volumetric flasks or calibrated 20mL disposable scintillation vials to prevent contamination of glassware.
- 10.7.1 To calibrate a 20mL scintillation vial, fill a 10mL volumetric flask with Hexane. Transfer this to the 20mL scintillation vial and mark the meniscus on the outside of the vial and discard the 10mL of hexane.
- 10.8 Place the flask or calibrated vial on the scale and tare. Then with a disposable pipette, transfer  $1\text{g} \pm 0.1\text{g}$  of each waste sample to the flask or vial. Repeat for all samples.
- 10.8.1 Choose a sample at random to perform a MS/MSD analysis and repeat section 10.8 for each MS/MSD sample.
- 10.8.2 If any sample has more than one phase, section 10.8 and all following procedures must be repeated for each phase.
- 10.9 Add at least 5mL of hexane to each sample and MS/MSD flask or vial then spike with 1mL of appropriate Surrogate Standard or QC Spikes.
- 10.10 Bring all samples and MS/MSD samples to 10mL final volume in the flasks or vials.
- 10.11 Transfer any sample or QC sample that is not already in a 20mL scintillation vial to a 20mL scintillation vial from the 10mL volumetric flask.
- 10.12 After all samples and QC have been transferred to 20mL scintillation vials, add 2g of baked sodium sulfate to each then cap and shake each vial for 2 minutes to remove any moisture from the sample.
- 10.13 After shaking for 2 minutes, transfer at least 5mL of each sample with a disposable pipette to a new 10mL scintillation vial or 10mL culture tube being careful not to transfer any sodium sulfate or water that may be present.
- 10.13.1 If necessary, the disposable pipette may be loosely packed with 2-3cm of baked glass wool to help further filter the waste dilution if high amounts of particulate solids are present.
- 10.14 At this point, the diluted waste samples may be transferred to autosampler vials and analyzed by GC/ECD.
- 10.14.1 It is highly recommended that waste dilution samples be further diluted by factors of 10 prior to analysis to prevent instrument contamination. If the sample has dark coloration, it should be diluted further until it is clear and then ran at increasing dilutions until the most optimal QC results may be obtained without contaminating the instrument.
- 11 Calculations**
- 11.1 Refer to GA EPD Laboratory SOP – Organochlorine Pesticides in Waste by Gas Chromatography – EPA Method SW846-8081A, SOP 1-034, online revision for calculations.

- 11.2 Refer to GA EPD Laboratory SOP – Polychlorinated Biphenyls (PCBs) in Waste by Gas Chromatography – EPA Method SW846-8082, SOP 1-036, online revision for calculations.

## **12 Waste Management**

- 12.1 See GA EPD Laboratory SOP-EPD Laboratory Waste Management Standard Operating Procedures, SOP6-015, online revision.

## **13 References**

- 13.1 GA EPD Laboratory SOP's - Initial Demonstration of Capability SOP 6-001, online revision and/or Continuing Demonstration of Capability SOP 6-002, online revision.
- 13.2 GA EPD Laboratory SOP – Spike Witnessing in the Organics Laboratory, SOP 1-044, online revision.
- 13.3 GA EPD Laboratory SOP – SOP for Muffle Furnace Baking of Sodium Sulfate, Glass Wool, Sodium Chloride and Sand, SOP 1-051, online revision.
- 13.4 GA EPD Laboratory SOP – Organochlorine Pesticides in Waste by Gas Chromatography – EPA Method SW846-8081A, SOP 1-034, online revision.
- 13.5 GA EPD Laboratory SOP – Polychlorinated Biphenyls (PCBs) in Waste by Gas Chromatography – EPA Method SW846-8082, SOP 1-036, online revision.
- 13.6 EPA Method SW846-8000B – Determinative Chromatographic Separations, Rev. 2, December 1996.
- 13.7 EPA Method SW846-8081A – Organochlorine Pesticides by Gas Chromatography, Rev. 1, December 1996.
- 13.8 EPA Method SW846-8082 – Polychlorinated Biphenyls (PCBs) By Gas Chromatography, Rev. 0, December 1996.
- 13.9 EPA Method SW846-3580A – Waste Dilution, Rev. 1, July 1992

## **14 Reporting Limits (RLs), Precision and Accuracy Criteria, and Quality Control Approach**

- 14.1 Refer to GA EPD Laboratory SOP – Organochlorine Pesticides in Waste by Gas Chromatography – EPA Method SW846-8081A, SOP 1-034, online revision.
- 14.2 Refer to GA EPD Laboratory SOP – Polychlorinated Biphenyls (PCBs) in Waste by Gas Chromatography – EPA Method SW846-8082, SOP 1-036, online revision.

## **15 Associated Labworks Test Codes**

- 15.1 Refer to GA EPD Laboratory SOP – Organochlorine Pesticides in Waste by Gas Chromatography – EPA Method SW846-8081A, SOP 1-034, online revision.

- 15.2 Refer to GA EPD Laboratory SOP – Polychlorinated Biphenyls (PCBs) in Waste by Gas Chromatography – EPA Method SW846-8082, SOP 1-036, online revision.

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